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(तीसरा पुनरीक्षण)

Indian Standard

TITANIUM DIOXIDE, ANATASE, FOR PAINTS — SPECIFICATION

(Third Revision)

UDC 667·622·118·22

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 1991

Price Group 3

Raw Materials for Paints Industry Sectional Committee, CHD 21

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paints Industry Sectional Committee had been approved by the Chemical Division Council.

This standard was first issued in 1953 and subsequently revised in 1968 and 1981. In the second revision, titanium dioxide pigment, rutile type was excluded and method for determination of relative density included. But at the same time relative density was not prescribed as the basis for a quantitative assay of two types of pigment, namely, anatase and rutile. Further, titanium dioxide content was increased from 96 to 97·5 percent by mass. The standard also achieved substantial alignment with ISO 591 : 1977 'Titanium dioxide for paints' issued by the International Organization for Standardization (ISO).

Though the two types of titanium dioxide pigment are identical in chemical composition, they differ in crystal structure and other physical properties. The determination of relative density provides a useful indication for distinguishing the two types, but X-ray diffraction is the only method which can clearly and conclusively identify the two types. The committee responsible for formulation of this standard has, therefore, decided to include X-ray diffraction method in this revision (*see* Annex A). Further, the tolerance for oil absorption of the approved sample has been changed from ± 5 to ± 10 percent.

The rutile type has separately been covered in IS 9788 : 1981 'Specification for titanium dioxide, rutile, for paints'.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retainded in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 JUNE 2003
TO
IS 411 : 1991 TITANIUM DIOXIDE, ANATASE, FOR PAINTS — SPECIFICATION

(Third Revision)

(*Page* 4, *Annex* B, *clause* **B-0**) — Substitute the following for the existing clause:

B-0 GENERAL

Three methods for determination of titanium dioxide have been prescribed In case of dispute, Method 1 shall be applicable.'

(*Page* 6, *Annex* B, *clause* **B-2.4**) — Insert the following new clause after **B-2.4**:

'B-3 METHOD 3 : GRAVIMETRIC METHOD

B-3.0 Outline of the Method

This method is applicable for determination of titanium dioxide content for anatase as well as rutile. Also, this method may be used to determine titanium dioxide content in pigments extracted from the paint where interference of elements like iron, zirconium, vanadium, molybdenum, tungsten, tin, antimony and bismuth is absent.

B-3.1 Principle

Titanium dioxide is dissolved in concentrated sulphuric acid in presence of ammonium sulphate. The undissolved siliceous matter is filtered. The filtrate is made alkaline with ammonia to precipitate titanium as titanium hydroxide. The precipitate is dissolved in hydrochloric acid and re-precipitated selectively through complexing with cupferron reagent. The complex is separated by filtration and then ignited to residue as titanium dioxide.

B-3.2 Apparatus

B-3.2.1 *Glass Beaker*, 500 ml and 1 litre.

B-3.2.2 *Watch Glass*

B-3.2.3 *Silica Crucible*, 50 ml.

B-3.2.4 *Desiccator*

B-3.2.5 *Electronic Balance,* accuracy up to 1 mg.

B-3.2.6 *Hot Plate.*

B-3.2.7 *Muffle Furnace*

B-3.3 Reagents

B-3.3.1 *Concentrated Sulphuric Acid*

B-3.3.2 *Ammonium Sulphate*

B-3.3.3 *Hydrochloric Acid Solution,* 1:1 diluted with water.

B-3.3.4 *Ammonia Solution,* 1:1 diluted with water.

B-3.3.5 2 *Percent Aqueous Solution of Ammonium Chloride*

B-3.3.6 3 *Percent Aqueous Solution of Cupferron Reagent,* freshly prepared and filtered.

B-3.3.7 *Methyl Orange Indicator*, 1% in alcohol.

B-3.4 Procedure

Weigh about 0.2 g of titanium dioxide sample to nearest 1 mg accurately into a 500-ml beaker, add 25 ml of concentrated sulphuric acid and 20 g of ammonium sulphate. Cover the beaker with a watch glass and heat strongly over a hot plate until a clear yellowish solution is obtained. This process takes about 30 min and is performed in a fume-cupboard. On cooling, the solution becomes colourless. Dilute the solution with 300 ml distilled water cautiously. Warm the solution and filter through a Whatman No. 40 filter paper in a 1 l beaker. To the filtrate, add a few drops of methyl orange indicator and make the solution alkaline by addition of 1:1 ammonia solution till yellow colour is observed. Boil the solution and filter through Whatman No. 41 filter paper and collect the precipitate of titanium hydroxide. Wash the precipitate with 1 percent solution of ammonia and transfer to a 500-ml beaker. Dissolve the precipitate in 100 ml of 1:1 hydrochloric acid and warm the solution. After warming dilute to 300 ml with distilled water; cool to 15°C. Treat the solution with 50 m of filtered aqueous solution of 3 percent cupferron reagent to give yellow flocculant precipitate.

Filter the precipitate through filter paper No. 41 (Whatman) and wash the precipitate with 2 pecent ammonium chloride solution.

Place the precipitate along with filter paper in a Previously dried and weighed crucible and heat on hot plate to dry. Ignite and incinerate the dried precipitate in a muffle furnace initially at 200°C for half an hour and raise the temperature within 600 to 700°C and maintain for an hour. Cool the residue in desiccator and weigh. Heat and cool the residue till a constant mass is obtained.

B-3.5 Calculation

$$\text{Titanium dioxide, percent} = \frac{\text{Mass of the residue} \times 100}{\text{Mass of the sample}}$$

(CHD 20)

Indian Standard

TITANIUM DIOXIDE, ANATASE, FOR PAINTS — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for titanium dioxide, anatase, used as a pigment in the paint industry.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
33 : 1990	Methods of sampling and test for inorganic pigments and extenders for paints (*third revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
1303 : 1983	Glossary of terms relating to paints (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 1303 : 1983 shall apply.

4 REQUIREMENTS

4.1 Description

It shall be a soft, dry powder and shall consist essentially of titanium dioxide of anatase crystal structure. If agreed to between the purchaser and the manufacturer, it shall be determined by X-ray diffraction method as prescribed in Annex A.

4.2 Composition

The material, after drying, as given in **8** of IS 33 : 1990 to constant mass, shall contain not less than 97·5 percent of titanium dioxide when determined by the method prescribed in Annex B.

4.3 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Titanium Dioxide, Anatase for Paints

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in IS 33 : 1990
(1)	(2)	(3)	(4)
i)	Volatile matter at 105 ± 2°C percent by mass, *Max*	0·5	8
ii)	Residue on 45 micron IS sieve, percent by mass, *Max*	0·1	9
iii)	Oil absorption, percent by mass (*see* Note 1)	15 to 30	10
iv)	Colour	Close match to the approved sample	11
v)	Reducing power	Not interior to the approved sample	15
vi)	Relative density at 27°C	3·7 to 3·9	16
vii)	Matter soluble in water, percent by mass, *Max*	0·5	19 (*see* Note 2)
viii)	*p*H of pigment slurry (without filtering) at 27°C	6 to 8	21

NOTES

1 This shall, however, be within ±10 percent of the approved sample, if any.

2 When a clear filtrate is not obtained in the test procedure, it shall be centrifuged before separation and evaporation of filtrate to determine matter soluble in water.

5 PACKING AND MARKING

5.1 Packing

The material shall be suitably packed in moisture proof packing as agreed to between the manufacturer and the purchaser.

5.2 Marking

The packages shall be marked with the following particulars:

a) Name of the material,

b) Indication of the source of manufacture,

c) Mass of the material,

d) Batch No. or lot No. in code or otherwise, and

e) Month and year of manufacture.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed under **5** of IS 33 : 1990.

6.2 All the characteristics prescribed in this standard shall be tested on the composite sample.

6.3 Criteria for Conformity

The lot shall be declared as conforming to the requirements of this standard if all the test results on the composite sample satisfy the relevant requirements prescribed in **4**.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemical that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 4.1)

DETERMINATION OF TITANIUM DIOXIDE, ANATASE, CRYSTAL STRUCTURE, BY X-RAY DIFFRACTION

A-0 OUTLINE OF THE METHOD

The X-ray diffraction pattern obtained from a material is characteristic of that material. The intensity of a diffraction peak is entirely dependent upon the amount of that substance present and to a minor extent the peak intensity of the component is also dependent on the mass absorption co-efficient of other materials present. The intensity of diffraction maxima for anatase and rutile is measured by X-ray diffractometry. The intensity of the anatase peak is converted to anatase content relative to rutile and the rutile content is determined by difference. The X-ray diffraction measurement is made on single pigments, pigment mixtures, on films of pigmented coatings and on films prepared from liquid coatings if interfering materials are not present. When interfering materials are present, the pigment is separated from the resolubilized (or ignited) film or from the liquid coating and treated to isolate the titanium dioxide.

A-1 APPARATUS

A-1.1 X-Ray Diffractometer

The principle components of this instrument are: (a) X-ray generator, (b) Copper target X-ray tube, (c) Goniometer, (d) Detector, and (e) Electronic circuit panel and strip chart recorder.

A-1.2 Operating Conditions

The X-ray tube voltage and filament current and other settings are selected to record X-ray diffraction peaks of weak intensities.

A-1.2.1 *Nickel Filter*

A nickel filter should be used to remove Cu K beta radiation if a monochromator is not used. Cu K beta radiation will produce a diffraction line from the rutile phase of TiO_2 that appears at the same 2θ angle as the anatase analytical line.

A-1.3 Typical Apparatus Conditions

A-1.3.1 *High-Voltage Power Supply*

Select X-ray tube voltage, filament current, and other settings so that 0·1 percent anatase generates a signal four times the noise level.

A-1.3.2 *Detector*

Scintillation detector operating at optimum voltage.

A-1.3.3 *Pulse Height Analyzer*

Settings will depend on instrumentation used.

A-1.3.4 *Chart-Speed* — 12·5 mm/min.

A-1.3.5 *Goniometer Scanning Speed* — 1/4°/min.

A-1.3.6 *Time Constant* — 5.

A-1.3.7 *Scanning Range*, $2\theta = 28$ to 24°.

A-1.3.8 *Scale Factors*

For samples of low anatase content, a scale factor of 100 counts full scale is normally used for the $2\theta = 26$ to 24° range (anatase diffraction maximum), and a scale factor of 5 000 counts full scale is normally used for the $2\theta = 28$ to 26° range (rutile diffraction maximum). The scale factors may be changed depending on the level of anatase expected in the sample.

A-2 REAGENTS

A-2.1 Purity of Reagents

Untreated rutile and anatase grades of titanium dioxide pigments are used to make synthetic standards for calibration. The crystalline structures of the reagents must be 100 percent rutile and 100 percent anatase as determined by examining these materials by X-ray diffraction.

A-2.2 Mixing Reagents

A series of rutile standards of varying rutile content are prepared to cover the range of interest by thoroughly mixing known amounts of 100 percent rutile and 100 percent anatase together. The pigments are dispersed in a solvent, such as isopropyl alcohol and then mixed, followed by air drying. This dry cake is ground up using a mortar and pestle to ensure homogeneity.

A-3 INTERFERENCES

A-3.1 Calcium sulphate interferes but its effect is eliminated by chemical removal. It is desirable to ensure by analysis that any residual calcium sulphate is considerably lower than the level of anatase being sought. The insoluble residue after removal of calcium sulphate should be ignited above 700°C. Chrome yellow and the valentinite form of antimony trioxide also interfere if not removed. Excessive amounts of iron render analysis difficult due to increased background (*see* Note). Additives, such as antimony and zinc, and impurities, such as niobium and zirconium, are generally present in solid solution and thus would not have interfering diffraction peaks. Surface elements such as silica and alumina, do not interfere. Extreme differences in particle size between the anatase and rutile portions affect the results.

NOTE — Background scatter due to high iron levels in a sample may be reduced by use of a cobalt or molybdenum target tube in place of the copper target tube. The background may be eliminated for all practical purposes by use of a curved crystal monochromator equipped with a graphite crystal in conjuction with a copper target tube.

A-3.2 In the calculation of converting the ratio to percent, it is implicitly assumed that the sum of anatase and rutile is 100 percent an assumption normally made in TiO_2 pigment systems. Other materials present would interfere to the extent that they dilute the sample. The third polymorphic form of TiO_2, brookite, would have such an effect. However, it reportedly does not occur in commercial titanium dioxide pigments.

A-4 SAMPLE PREPARATION

A-4.1 Packing the pigment in the specimen holder, and obtaining a smooth surface is one of the most important phases in X-ray analysis. Ripples or indentures in the specimen surface cause variations in the test because of an error of eccentricity and a change in the intensities of the peaks. The change of preferred orientation of pigmentary TiO_2 is remote because of its small particle size.

A-4.1.1 Pack the pigment in a die suitably constructed to accommodate the specimen holder for goniometer. Apply constant pressure with a hydraulic press (*see* Note). Alternatively, place the specimen holder on a flat, smooth, firm surface and pack the specimen into the opening by applying constant and firm pressure with a flat blade.

NOTE — Use of a set procedure with a hydraulic press will improve specimen preparation uniformity compared to hand-packed specimens. This is particularly true where more than one person is involved in specimen preparation.

A-4.1.2 *Coating on Metal Panels*

Cut the coated panel into the proper shape and size to fit an adjustable specimen holder and analyze without further preparation. This assumes that interferences are not present.

A-4.2 Liquid Coating Samples

A-4.2.1 Liquid coating samples not containing interfering materials are analyzed as cast film of the total paint on aluminium or other suitable substrate. A 25 to 50 µm: (1 to 2 mil) dry film thickness is adequate.

A-4.2.2 Liquid paint samples containing interfering materials must not be analyzed until the interfering components are removed. This can be accomplished by centrifuging to separate the total pigment. The separated pigment is treated to remove interfering components.

A-5 PROCEDURE

A-5.1 Record the X-ray diffraction maxima of the anatase and of the rutile peak by scanning the range between $2\theta = 26$ to 24° and $2\theta = 28$ to 26°, respectively. Scan each range twice.

A-5.2 Measure the peak maxima in chart units for the anatase and rutile peaks. Draw the baseline between the lowest points of the trace on each side of the peak. Average the peak maxima of the duplicate runs and convert chart units into counts per second.

A-5.3 Since only ratios are used, the chart units need only be corrected for differences in the scale factors used for the two peaks. Any consistent units can be used, not just counts per second.

A-5.4 Prior to and after analyzing the unknown sample, analysis of one of the standards should be made to establish instrument stability over a period of time.

A-6 CALCULATION

A-6.1 Calculate the anatase level relative to rutile from the intensities above background as follows:

$$\text{Percent anatase} = \frac{1}{1 + K\dfrac{I_r}{I_a}} \times 100$$

where

I_r = net intensity of rutile diffraction maximum,

I_a = net intensity of anatase diffraction maximum, and

K = slope of calibration curve when intensity ratio I_a/I_r is plotted against mass ratio M_a/M_r and equals $(I_a/I_r)/(M_a/M_r)$.

where

M_a = mass or anatase, and

M_r = mass of rutile.

A-6.2 The value of the constant in the intensity-concentration equation is determined for individual instruments with standards in the concentration range of interest. Routine instrument-calibration procedures are followed.

ANNEX B

(*Clause* 4.2)

DETERMINATION OF TITANIUM DIOXIDE

B-0 GENERAL — Two methods for determination of titanium dioxide have been prescribed. For routine testing, either Method 1 or Method 2 may be followed, but in case of dispute, Method 1 shall be applicable.

B-1 METHOD 1 : ZINC REDUCTION METHOD

B-1.0 Outline of the Method

A solution of the dry pigment is made with concentrated sulphuric acid and ammonium sulphate. Titanium is reduced with zinc amalgam in Nakazono reductor and the resulting trivalent titanium is titrated with ferric alum.

B-1.1 Reagents

B-1.1.1 *Concentrated Sulphuric Acid* — Relative density 1·84.

B-1.1.2 *Ammonium Sulphate*

B-1.1.3 *Zinc Amalgam* — 3 percent. Place 50 ml of mercury in a small porcelain dish on a hot water-bath, covering the surface of mercury with 2 percent (*m/v*) sulphuric acid. Add about 25 g of zinc in small granules. Stir from time to time and replenish the dilute acid with water as required. When all the zinc has disappeared, allow the amalgam to cool and stand for several hours. Finally filter through a Gooch crucible with no asbestos pad. Store the amalgam in a glass bottle under 2 percent (*m/v*) sulphuric acid.

B-1.1.4 *Standard Ferric Ammonium Sulphate Solution* — 0·062 5 N. Standardize as given in **B-1.1.4.1**.

B-1.1.4.1 Transfer by a pipette 50 ml of the ferric ammonium sulphate solution to be standardized to the Nakazono reductor (*see* Fig. 1) prepared ready for use as described in **B-1.3.2.** Displace the air in the reductor by passing carbon dioxide for 3 minutes. Close the taps B and C. Bring the amalgam into intimate contact with ferric ammonium sulphate solution by shaking the apparatus vigorously for 5 minutes. Place the apparatus on a ring stand. Drain the amalgam and pass inert gas as prescribed under **B-1.3.4**. Titrate this against standard potassium permanganate solution. The end point is the appearance of light pink colour persistent for 30 seconds at least. Calculate the strength of the ferric ammonium sulphate solution as follows:

$$\text{Strength of ferric ammouium sulphate solution} = V \times \frac{0{\cdot}1}{50}$$

where

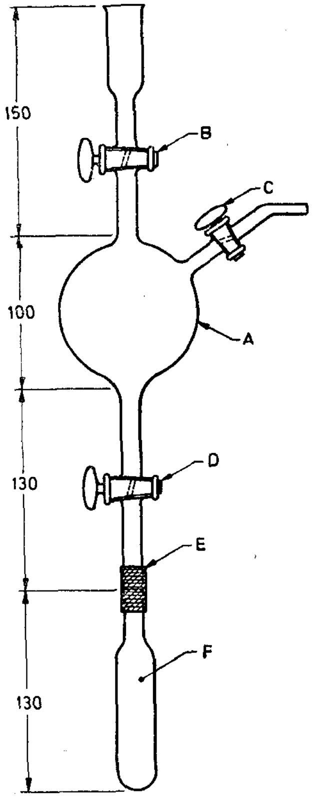
V = volume, in ml, of 0·1 N potassium permanganate solution required for titration.

B-1.1.5 *Potassium Thiocyanate Solution* — 10 percent (*m/v*).

B-1.2 Apparatus

B-1.2.1 *Nakazono Reductor*

The apparatus, as shown in Fig. 1, consists of a bulb *A* with a capacity of 500 ml with three stopcocks *B*, *C* and *D* attached. To the stopcock *D* may be attached, by means of thick rubber tubing *E*, a small cylindrical flask *F* of about 50 ml capacity. The stopcock *C* which is smaller in bore than either of the other two serves to admit carbon dioxide or nitrogen.



All dimensions in millimetres.

FIG. 1 NAKAZONO REDUCTOR

B-1.3 Procedure

B-1.3.1 Weigh approximately 0·5 g of the dried material to the nearest 0·1 mg and transfer to a 250-ml beaker. Add 20 ml of concentrated sulphuric acid and 10 g of ammonium sulphate. Mix carefully and heat on a hot plate to fuming and then over a strong flame until solution is complete, observing caution while examining the solution. Cool the solution, dilute with 100 ml of water, stir and filter, if necessary.

B-1.3.2 Open stopcock *C*, transfer sufficient quantity of 4 percent sulphuric acid to fill the bottom flask and leave no air space below the stopcock *D*. Close tap *D* and add 20 ml of zinc amalgam to *A*. The reductor is now ready for use.

B-1.3.3 To the amalgam contained in *A*, transfer the solution, warmed to 50°C, which shall not exceed 300 ml in volume. Then pass carbon dioxide or nitrogen for about 3 minutes. Close taps *B* and *C* and simultaneously disconnect the carbon dioxide supply. Bring the amalgam into intimate contact with the solution by shaking the apparatus vigorously for 5 minutes, holding it in such a way that the stopcocks are kept shut and in position. The rate of reduction is apparent from the development of the characteristic violet colour of the trivalent titanium.

B-1.3.4 Place the apparatus on a ring stand. Open stopcock *D* slightly and allow the amalgam to flow sowly into *F*. Close *D* immediately as soon as the amalgam has been completely transferred. With taps *B* and *D* open, pass inert gas through *D* as before, add 10 ml of potassium thiocyanate solution and titrate with standard ferric ammonium sulphate solution until a faint pink colouration persists for one minute.

B-1.3.5 Facilitate the titration by attaching a piece of very narrow glass tubing to the top of the burette with a piece of rubber tubing. The extension passes through stopcock *B* into the bulb *A*.

B-1.4 Calculation

B-1.4.1 Calculate the titanium dioxide content of the material as follows:

$$\text{Titanium dioxide content, percent by mass} = \frac{V \times 0{\cdot}005}{M} \times 100$$

where

V = volume, in ml, of standard ferric ammonium sulphate solution required, and

M = mass, in g, of pigment taken.

B-2 MTEHOD 2 : ALUMINIUM REDUCTION METHOD

B-2.0 Outline of the Method

This method is similar to zinc reduction method but easier and quicker to operate. Titanium is reduced to trivalent state by aluminium and titrated against ferric ammonium sulphate using ammonium thiocyanate as indicator.

B-2.1 Apparatus

B-2.1.1 *Delivery Tube*

Made of about 4 mm internal diameter glass tube bent in such a way as to have a horizontal run of about 150 mm and vertical drop of about 75 mm at one end, and a vertical drop of about 150 mm at the other end.

B-2.1.2 *Long Necked Flask* — 500-ml capacity.

B-2.2 Reagents

B-2.2.1 *Ammonium Sulphate*

B-2.2.2 *Sulphuric Acid* — relative density 1·84.

B-2.2.3 *Hydrochloric Acid* — relative density 1·19.

B-2.2.4 *Aluminium Foil* — electrolytic grade.

B-2.2.5 *Sodium Bicarbonate Solution* — saturated.

B-2.2.6 *Ammonium Thiocyanate Indicator Solution* — Prepare by dissolving 24·5 g of ammonium thiocyanate in 80 ml of hot water. Filter, cool to room temperature, dilute to 100 ml and preserve in well stoppered, amber coloured bottle.

B-2.2.7 *Standard Ferric Ammonium Sulphate Solution* — Prepare by dissolving 30·16 of fresh ferric ammonium sulphate [$FeNH_4(SO_4)_2, 12\,H_2O$] in 800 ml of water containing 15 ml of concentrated sulphuric acid. Add 0·1 N potassium permanganate solution until a very slight pink colour is obtained. Dilute to 1 000 ml and mix thoroughly. Filter, if necessary. Standardize with 0·190 to 0·210 g of standard titanium dioxide as prescribed in **B-2.3** and calculate titanium dioxide equivalent of solution in grams of titanium dioxide per ml of solution as given below:

$$\text{Titanium dioxide equivalent} = \frac{M \times P}{V \times 100}$$

where

M = mass, in g, of standard titanium dioxide taken for test,

P = percentage of titanium dioxide in standard dioxide used, and

V = volume, in ml, of ferric ammonium sulphate solution required for filtration.

B-2.3 Procedure

B-2.3.1 Weigh accurately 0·190 to 0·210 g of the dry material and transfer the same to a 500-ml long necked flask. Add 7 to 9 g of ammonium sulphate and 20 ml of sulphuric acid. Mix carefully, heat on a hot plate to fuming and then over a strong flame until solution is complete. Cool and add 120 ml of water and 20 ml of hydrochloric acid. Boil the contents and remove from heat.

B-2.3.2 Insert the short end of the delivery tube into one hole of a two-hole rubber stopper suitable for the long necked flask. Insert a glass rod with a hook at the bottom end in the other hole in such a way that the bottom end shall be nearer to the bottom of the flask when the stopper is fitted into the flask. Attach 1 g of aluminium foil to the bottom end of the rod by coiling it around the rod. Insert the stopper carrying the rod with foil and delivery tube into the flask in such a way that the foil is near the bottom of the flask at the same time that the long end of the delivery tube is near the bottom of a 250-ml beaker containing about 150 ml of sodium bicarbonate solution.

B-2.3.3 Heat the flask as soon as the dissolution of aluminium is complete to gentle boiling for 3 to 5 minutes without disturbing the assembly. Cool to 60°C by partial immersion in water and siphon the sodium bicarbonate solution into the flask giving an atmosphere of carbon dioxide over the reduced titanium solution. Withdraw the stopper, rinse the glass rod attached to it with a little of water, catching the rinsings in the flask. Add 2 ml of ammonium thiocyanate indicator solution and titrate immediately against standard ferric ammonium sulphate solution to a straw coloured end point.

B-2.4 Calculation

B-2.4.1 Calculate the percentage of titanium dioxide as follows:

$$\text{Titanium dioxide, percent} = \frac{V \times E \times 100}{M}$$

where

V = volume, in ml of ferric ammonium sulphate solution used in test,

E = titanium dioxide equivalent (*see* **B-2.2.7**) of ferric ammonium sulphate solution in g/ml, and

M = mass, in g, of material taken for test.

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